

REPORT DOCUMENTATION PAGE

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13. ABSTRACT (Maximum 200 Words) This document reports on the final disposition of a \$220,000 project supported by AFOSR for instrumentation to construct an interfacial chemistry and electrochemistry facility residing in the Department of Chemistry and the Department of Materials Science and Engineering at Ohio State University under project no. F49620-00-1-0274. This grant was awarded in March, 2000. A matching amount equal to \$220,000 was made by the Ohio Board of Regents, the Colleges of engineering, and Math and Physical Sciences, as well as the Departments of Materials Science and Engineering and Chemistry to bring the total award to \$440,000. Funds were fully expended by November, 2000 on the following instrumentation: Phase identification and imaging system, Process Raman spectrometer, High performance potentiostat, Nd:YAG CW laser, Scanning probe microscope station, Precision surface preparation tool, Slow strain rate test system, Rotating ring disk electrode electrochemical detection system. All of this equipment is now in place and operational and is being used in support of several AFOSR-sponsored research programs.				
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Final Project Report

Corrosion and Coatings Research Facility
(a request for instrumentation)

Program contract no. F49620-00-1-0274

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Overview

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- Phase identification and imaging system
- Process Raman spectrometer
- High performance potentiostat
- Nd:YAG CW laser
- Scanning probe microscope station
- Precision surface preparation tool
- Slow strain rate test system
- Rotating ring disk electrode electrochemical detection system

All of this equipment is now in place and operational and is being used in support of several AFOSR-sponsored research programs.

Equipment purchased under this project is detailed below.

Bruker Equinox 55 FTIR spectrometer.

This FTIR spectrometer is used to examine thin films on metal surfaces, including conversion coatings and corrosion inhibitor candidates. More commonly, it is used with a FT Raman module to acquire Raman spectra with a 1064 nm laser. For samples which exhibit fluorescence, the longer laser wavelength leads to higher quality Raman spectra. In addition, chromate oxyanions are resonance enhanced and 1064 nm excitation can avoid resonance and permit observation of weaker features. A good example of the benefit of 1064 nm excitation is observation of the Cr-O-Cr bridging mode in $\text{Cr}_2\text{O}_7^{2-}$ (J. D. Ramsey, L. Xia, M. W. Kendig, McCreery, R. L. "Raman spectroscopic analysis of the speciation of dilute chromate solutions", *Corros. Sci.*, 2001, 43 (8), 1557-1572).

Perkin Elmer Lambda 900 UV-Vis-NIR spectrometer.

This spectrometer includes several specular and diffuse reflectance attachments for observing thin films on metal surfaces. One goal is to observe the early stages of coating formation, and to observe inhibitor interactions with metal surfaces. Of particular interest is Cr^{III} oxy-hydroxide, which is difficult to observe with Raman. It is likely to be the immediate product of Cr^{VI} reduction on aluminum alloys, and may be the active inhibitor of O_2 reduction.

Perkin Elmer Lambda EZ201 UV-Vis spectrometer.

A routine UV-Vis spectrometer for monitoring dilute inhibitors or corrosion products in solution. Intended for simple transmission experiments, particularly those involving release of inhibitors from coatings on metals, using the "artificial scratch" cell. An example is such measurements is: L. Xia, E. Akiyama, G. Frankel, R.L. McCreery, "Storage and Release of Soluble Hexavalent Chromium from Chromate Conversion Coatings", *J. Electrochem. Soc.*, 2000, 147, 2556 - 2562.

Bioanalytical Systems Epsilon Computer controlled potentiostat.

A conventional potentiostat with low current capability, for cyclic voltammetry, polarization curves, and sample pretreatment. The techniques and data presentation of this system complement those of the Gamry systems made specifically for corrosion experiments.

Scanning Probe Microscope.

A Veeco/Digital Instruments Dimension 3100 was purchased with a cantilever holder for scanning in liquids and an application module that enables the instrument to be used for Scanning Kelvin Probe Force Microscopy. This instrument also has a 6 in vacuum chuck that permits the study of large samples. This instrument has allowed us to continue the studies of Al alloy corrosion and inhibition by AFM-based methods. In particular, we have used the instrument recently to do masking experiments in which a protective polymeric ink layer is deposited on the surface, and windows are opened up by AFM scratching to expose different areas of the microstructure. Such samples provide information regarding the behavior of the different constituents of the AA2024 matrix. The easy sample positioning capability and optical system associated with the new scope made this work possible.

Struers Precision Surface Preparation Tool

A high precision polishing unit was purchased to prepare surfaces for examination by the DI Dimension 3100 scanning probe microscope.

Rotating Ring Disk Electrode

A Model 636 Rotating Ring Disk Electrode system was purchased from Perkin Elmer Princeton Applied Research Electrochemical Instruments Division. This instrument is capable of operating in either a rotating disk or rotating ring-disk configuration. It is capable of rotational speeds up to 10,000 RPM, and has a quick change electrode capability which is well suited for corrosion studies.

Environmental Fracture System

To support environmental cracking studies, a constant extension rate load frame was purchased and an alternate immersion tank was constructed. An M-CERT™ constant extension rate test system was obtained from Intercorr International, Inc. The frame is rated for a 44,000 N load capacity, total stroke of 2 inches, and is capable of producing strain rates ranging from 10^{-7} to 10^{-3} s⁻¹.

The system has been modified to conduct aqueous environmental exposures under free corrosion conditions and under potentiostatic or galvanostatic control. The system is currently under use for evaluation of environmental cracking susceptibility for Al-Mg alloys, friction stir welded Al-Zn-Mg-Cu alloys, and Al-Li-Cu alloys under development by the Air Force.

Noran Phase ID system.

This phase identification (phase ID) accurately identifies crystalline compounds during scanning electron microscopy. The technique is based on electron backscatter diffraction, which produces a Kikuchi pattern that uniquely identifies the compound. By combining this diffraction information with elemental information derived from energy dispersive spectroscopy, it is possible to rapidly and accurately identify crystalline materials using a search match routine with a comprehensive database. The Phase ID system installed is state-of-the-art instrumentation, which enables quantitative structural and chemical analysis of heterogeneous samples with sub-micron resolution. This equipment has been added on to an existing Philips XL-30 SEM in the Campus Electron Optics Facility.